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W. Small IV, C. T. Alviso, T. S. Wilson

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Work Done By: C.T. Alviso (aging), W. Small (tensile testing and analysis)
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Report Written By: W. Small
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SUMMARY

TR-55 silicone rubber samples were previously subjected to an aging process consisting of the application of a tensile strain of approximately 0, 67, 100, 133, or 167% elongation (axial stretch ratio λ_1 = 1.00, 1.67, 2.00, 2.33, or 2.67) during exposure to gamma radiation doses of 0, 5, 10, or 17 Mrad under vacuum. Two rectangular specimens were cut from each sample for uniaxial tensile testing. Each specimen was subjected to four load/unload cycles followed immediately by strain to failure and stress-strain curves were generated. The Young's (elastic) modulus was calculated based on the initial slope of the stress-strain curve of the first loading segment. Tensile strength and failure strain were determined.

Mechanical behavior was dependent on the aging elongation and radiation dose. All specimens showed a large hysteresis loss during the first load/unload cycle. Subsequent cycles showed stress softening characteristic of the Mullins effect with much smaller hysteresis losses. Mullins effect behavior was also evident in the first loading segment of strain aged specimens, which showed an initial period of stress softening dependent on the aging elongation. Also, higher elongation resulted in higher stress at the end of the first loading segment. The peak load stress decreased with each cycle for all specimens. Residual plastic deformation was evident after the first cycle. The modulus increased with radiation dose, while the tensile strength and failure strain decreased. Elongation during irradiation generally resulted in lower values of the modulus, tensile strength, and failure strain compared to unstrained specimens.

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METHODS

The TR-55 material was made at Kansas City Plant. The samples were previously subjected to an aging process consisting of the application of a tensile strain of approximately 0, 67, 100, 133, or 167% elongation during exposure to gamma radiation doses of 0, 5, 10, or 17 Mrad under vacuum. In terms of the axial stretch ratio, λ_1 was nominally 1.00, 1.67, 2.00, 2.33, or 2.67 (actual elongation was generally slightly less than the nominal value due to slippage of the specimen in the stretching fixture). The strain aging process is described in detail in Appendix A. The samples were allowed to recover at room temperature without external stimuli before tensile testing.

Two rectangular specimens, each approximately 39 mm long, were cut from each sample using a razor blade immediately before testing. Specimen width was between 2.67 and 3.38 mm. Specimen thickness was between 0.577 mm and 0.910 mm depending on the extent of the permanent set induced by the aging elongation. Permanent set data was recorded following the aging process by C.T. Alviso and is not included in this report. Width and thickness measurements were made using a micrometer.

Uniaxial tensile testing was done on an Instron 5565 dual-column electromechanical test system equipped with pneumatic grips (N_2 pressure = 80 psi) and a 50-N load cell. Specimens were secured in the grips (initial grip separation ≈ 20 mm) and stretched at a rate of 20 mm/min (strain rate ≈ 1 min^{-1}). Each specimen was subjected to four load/unload cycles. The loading segment stretched the specimen to an engineering strain of 0.5, 1.0, 1.5, or 2.2 (see Table 1) and held it there for 5 s. The unloading segment returned the specimen to the original starting position and held it there for 5 s. The specimen was stretched to failure immediately after the fourth unloading segment. Data was acquired at a rate of 10 Hz. Stress was calculated from the load cell output. Strain was calculated from the crosshead position. All tests were performed at room temperature in the Associate Space in B153.

Simply securing the specimen in the grips resulted in a compressive load as the grips were tightened. To eliminate this initial compression of the specimen, the following procedure was performed. First, the grip separation was set to 20 mm. Second, the specimen was secured in the movable upper grip only. Third, the upper grip was lowered ~1 mm. Finally, the specimen was secured in the fixed lower grip, and the upper grip was raised until the load returned to $\pm 10^{-3}$ N. The grip separation was slightly different from 20 mm following this procedure. Because the actual initial grip separation was used to calculate the strain, the calculated initial strain was zero.

The Young's (elastic) modulus was given by the slope of a line fit to the true stress-strain data between 0 and 0.02 strain. Using the calculated modulus, Hookean and neo-Hookean models were used to calculate the theoretical stress at low strain for comparison to the experimental stress-strain data. Formulas used to calculate stress and strain, and the Hookean and neo-Hookean models, are given in Appendix B.

RESULTS

Young's modulus, failure strain, and failure stress (i.e., tensile strength) for all specimens are given in Table 1. Average values for the two specimens are given in Table 2 and Fig. 1. The modulus increased with radiation dose, while the tensile strength and failure strain decreased. Elongation during irradiation generally resulted in lower values of the modulus, tensile strength, and failure strain compared to unstrained specimens.

True stress-strain curves of the first loading segment at low (<0.16) strain are compared to theoretical Hookean and neo-Hookean curves in Fig. 2. At strains below 0.05, both models are in good agreement with the data, indicating essentially Hookean behavior at very low strain for virgin and aged specimens. At slightly higher strains up to 0.10, the Hookean model is generally in better agreement with the data for the unstrained irradiated specimens, while the neo-Hookean model is generally better for the virgin specimens and specimens strained during irradiation.

True stress-strain curves of the first loading segment are shown in Fig. 3. Each plot includes a curve corresponding to the virgin material for reference. Mullins effect behavior is evident in the first loading segment of strain aged specimens, which showed an initial period of stress softening dependent on the elongation; higher elongation yielded a longer period of stress softening. Also, higher elongation resulted in higher stress at the end of the loading segment.

True stress-strain curves of the four load/unload cycles and subsequent strain to failure are shown in Fig. 4. In Fig. 5, the strain to failure curve has been excluded and each cycle is shown in a different color to better distinguish each cycle. All specimens showed a large hysteresis loss during the first load/unload cycle (the stress was lower during unloading). The second cycle showed stress softening characteristic of the Mullins effect with much smaller hysteresis loss. Very little additional softening occurred during extension up to the same maximum strain in subsequent cycles. During the final pull, the stress was reduced up to the previous maximum strain, then increased to rejoin the first load curve. The peak load stress decreased with each cycle for all specimens. Residual plastic deformation was evident after the first cycle.

Table 1: Results of tensile testing

			Aging Conditions			Specimen Dimensions		Calculated Mechanical Properties		
Test Date	Specimen	Cyclic Eng Strain	Atmosphere	**Irradiation**	**Elongation**	Width	Thickness	E (Young's Mod)	Failure True Strain	Failure True Stress
				(Mrad)	**(%)**	(mm)	(mm)	(MPa)		(MPa)
4/19/2010	1	2.20	air	**0**	**0**	2.99	0.885	4.76	not determined	not determined
4/19/2010	2	2.20	air	**0**	**0**	3.27	0.887	4.82	>2.32	>75.17
			air	**0**	**67**					
			air	**0**	**67**					
			air	**0**	**100**					
			air	**0**	**100**					
			air	**0**	**133**					
			air	**0**	**133**					
			air	**0**	**167**					
			air	**0**	**167**					
4/16/2010	1	0.50	vac	**5**	**0**	3.05	0.910	5.85	1.84	57.64
4/19/2010	2	1.50	vac	**5**	**0**	3.38	0.907	5.05	1.79	45.43
4/16/2010	1	0.50	vac	**5**	**67**	3.14	0.811	3.83	1.64	47.56
4/19/2010	2	1.50	vac	**5**	**67**	3.31	0.814	3.87	1.46	33.56
4/16/2010	1	0.50	vac	**5**	**100**	3.05	0.775	3.96	1.49	41.02
4/19/2010	2	1.50	vac	**5**	**100**	3.12	0.762	3.12	1.40	33.43
4/16/2010	1	0.50	vac	**5**	**133**	2.95	0.750	3.82	1.25	27.19
4/19/2010	2	1.50	vac	**5**	**133**	3.34	0.721	3.62	1.58	47.69
4/16/2010	1	0.50	vac	**5**	**167**	3.10	0.744	3.55	1.29	32.14
4/19/2010	2	1.50	vac	**5**	**167**	2.89	0.734	3.42	1.54	52.09
4/16/2010	1	0.50	vac	**10**	**0**	3.11	0.864	7.49	1.28	28.98
4/16/2010	2	1.00	vac	**10**	**0**	3.07	0.852	7.02	1.31	30.46
4/16/2010	1	0.50	vac	**10**	**67**	2.99	0.719	5.94	0.90	19.06
4/16/2010	2	1.00	vac	**10**	**67**	3.15	0.731	5.78	0.93	20.42
4/16/2010	1	0.50	vac	**10**	**100**	3.15	0.723	5.34	0.94	22.83
4/16/2010	2	1.00	vac	**10**	**100**	3.32	0.705	5.92	0.94	22.58
4/16/2010	1	0.50	vac	**10**	**133**	3.26	0.661	5.80	0.85	21.81
4/16/2010	2	1.00	vac	**10**	**133**	3.10	0.661	5.43	0.80	18.72
4/16/2010	1	0.50	vac	**10**	**167**	2.67	0.637	5.52	1.04	36.45
4/16/2010	2	1.00	vac	**10**	**167**	3.01	0.651	5.23	0.89	24.81
4/15/2010	1	0.50	vac	**17**	**0**	3.17	0.860	8.55	0.91	18.49
4/16/2010	2	0.50	vac	**17**	**0**	2.98	0.855	8.22	0.79	14.31
4/15/2010	1	0.50	vac	**17**	**67**	3.31	0.646	8.46	0.63	14.02
4/16/2010	2	0.50	vac	**17**	**67**	3.05	0.648	8.65	0.65	15.96
4/15/2010	1	0.50	vac	**17**	**100**	3.16	0.645	8.57	0.58	15.49
4/16/2010	2	0.50	vac	**17**	**100**	3.00	0.653	8.45	0.56	14.46
4/15/2010	1	0.50	vac	**17**	**133**	3.10	0.595	7.78	0.58	17.02
4/16/2010	2	0.50	vac	**17**	**133**	3.28	0.577	8.20	0.59	18.05
4/15/2010	1	N/A	vac	**17**	**167**	2.82	0.618	7.96	0.53	16.15
4/15/2010	2	0.50	vac	**17**	**167**	2.92	0.624	7.97	0.64	21.45

Note: One specimen (17 Mrad, 167% elongation, Specimen 1) was not cycled prior to straining to failure.

Table 2: Average values (n=2) of Young's modulus, failure strain, and tensile strength

	0%		67%		100%		133%		167%	
	Avg	StDev	Avg	StDev	Avg	StDev	Avg	StDev	Avg	StDev
Young's Modulus (MPa)										
0 Mrad	4.79	0.05	-	-	-	-	-	-	-	-
5 Mrad	5.45	0.57	3.85	0.03	3.54	0.59	3.72	0.14	3.49	0.09
10 Mrad	7.26	0.33	5.86	0.11	5.63	0.41	5.61	0.26	5.37	0.20
17Mrad	8.38	0.23	8.55	0.14	8.51	0.09	7.99	0.30	7.97	0.01
Failure Strain (True)										
0 Mrad	-	-	-	-	-	-	-	-	-	-
5 Mrad	1.81	0.04	1.55	0.13	1.45	0.06	1.41	0.23	1.42	0.18
10 Mrad	1.30	0.02	0.91	0.02	0.94	0.00	0.83	0.04	0.96	0.11
17Mrad	0.85	0.08	0.64	0.02	0.57	0.02	0.59	0.01	0.58	0.07
Tensile Strength (True) (MPa)										
0 Mrad	-	-	-	-	-	-	-	-	-	-
5 Mrad	51.53	8.63	40.56	9.90	37.23	5.37	37.44	14.50	42.12	14.10
10 Mrad	29.72	1.05	19.74	0.96	22.71	0.17	20.27	2.19	30.63	8.24
17Mrad	16.40	2.95	14.99	1.38	14.98	0.72	17.53	0.73	18.80	3.75

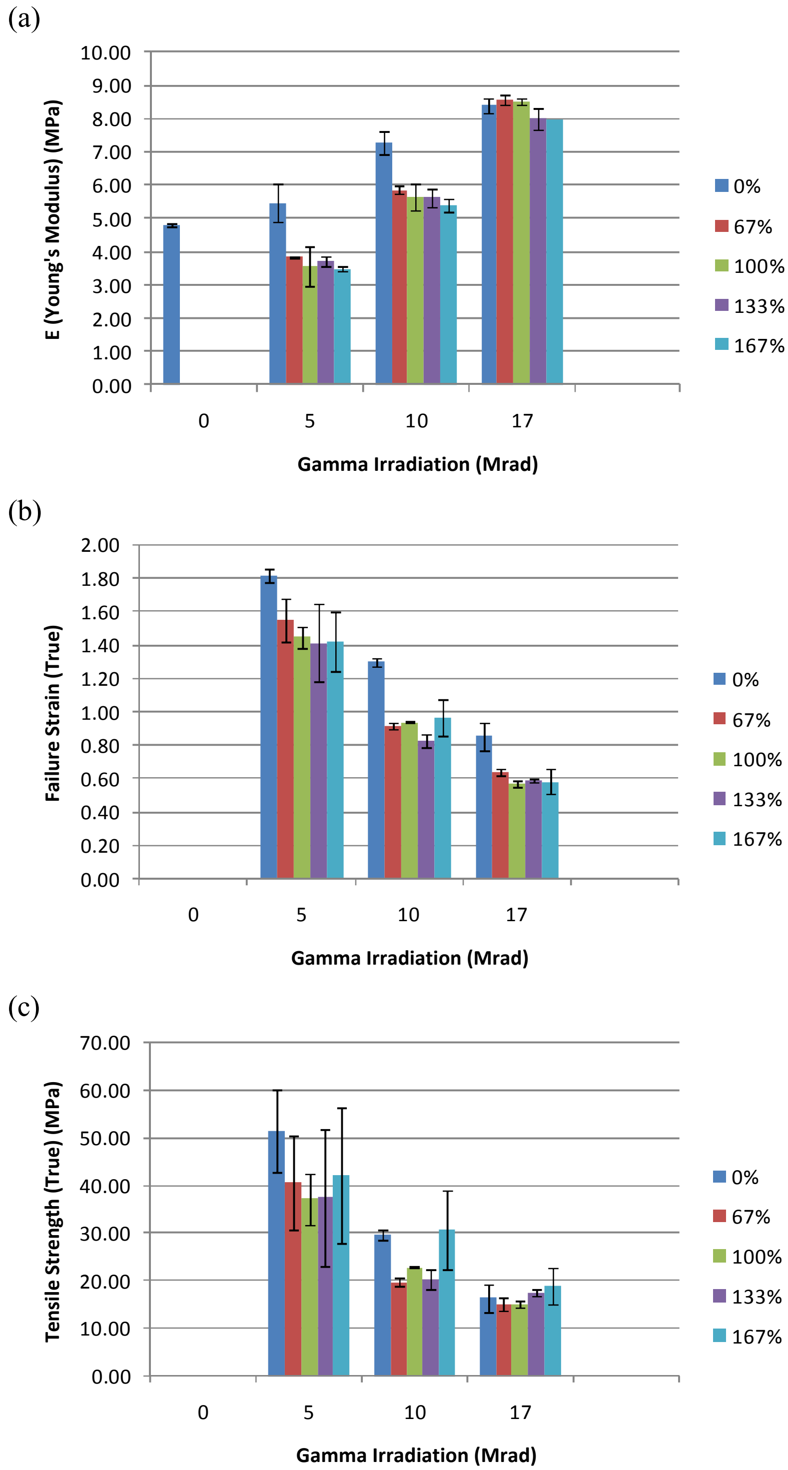

Fig. 1. Young's modulus, failure strain, and tensile strength for TR-55 specimens irradiated under tensile strain. The bars are average values (n=2) and the error bars are standard deviation.

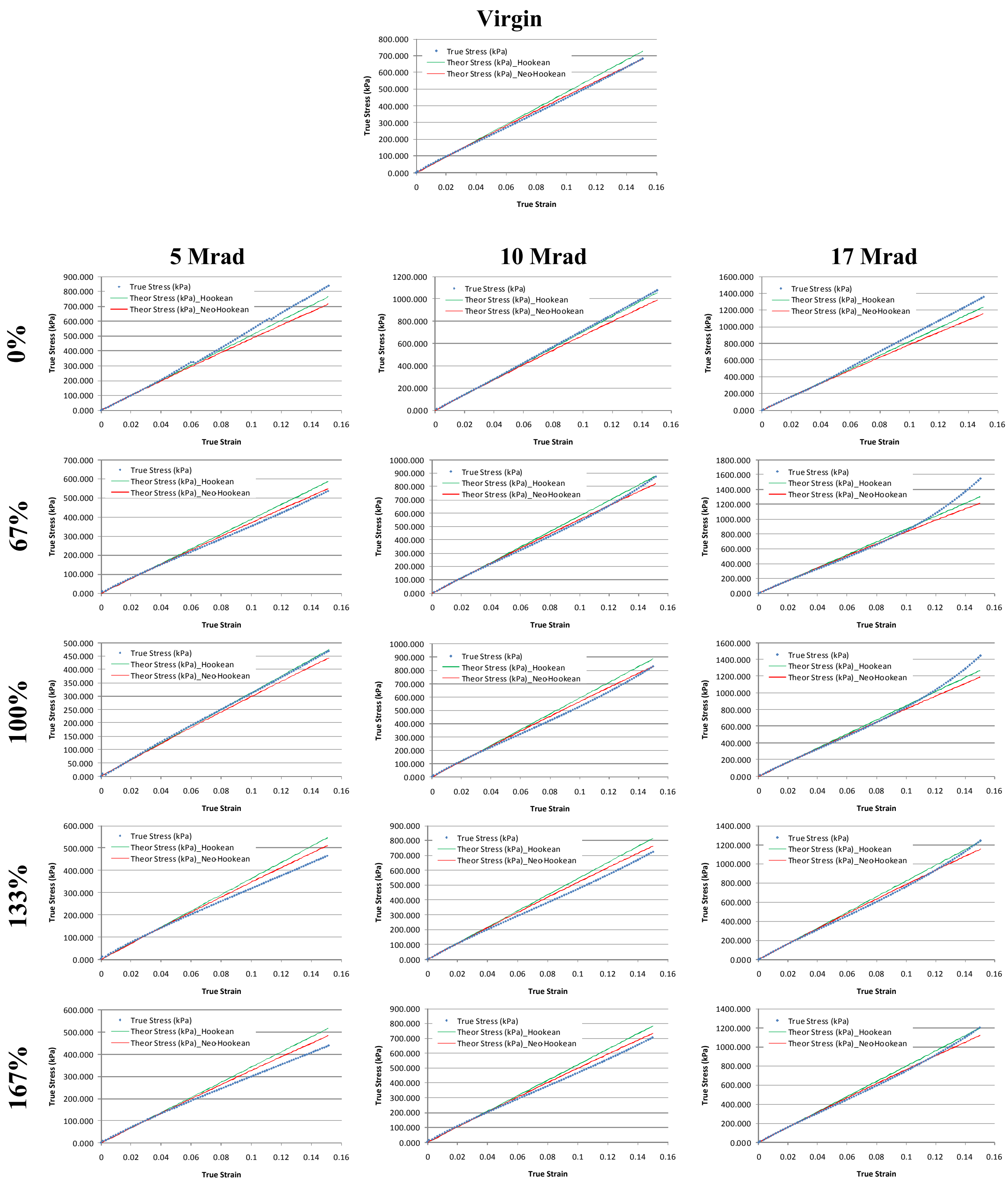

Fig. 2. Experimental and theoretical (Hookean and neo-Hookean) stress at low strain. Specimen 2 data is shown.

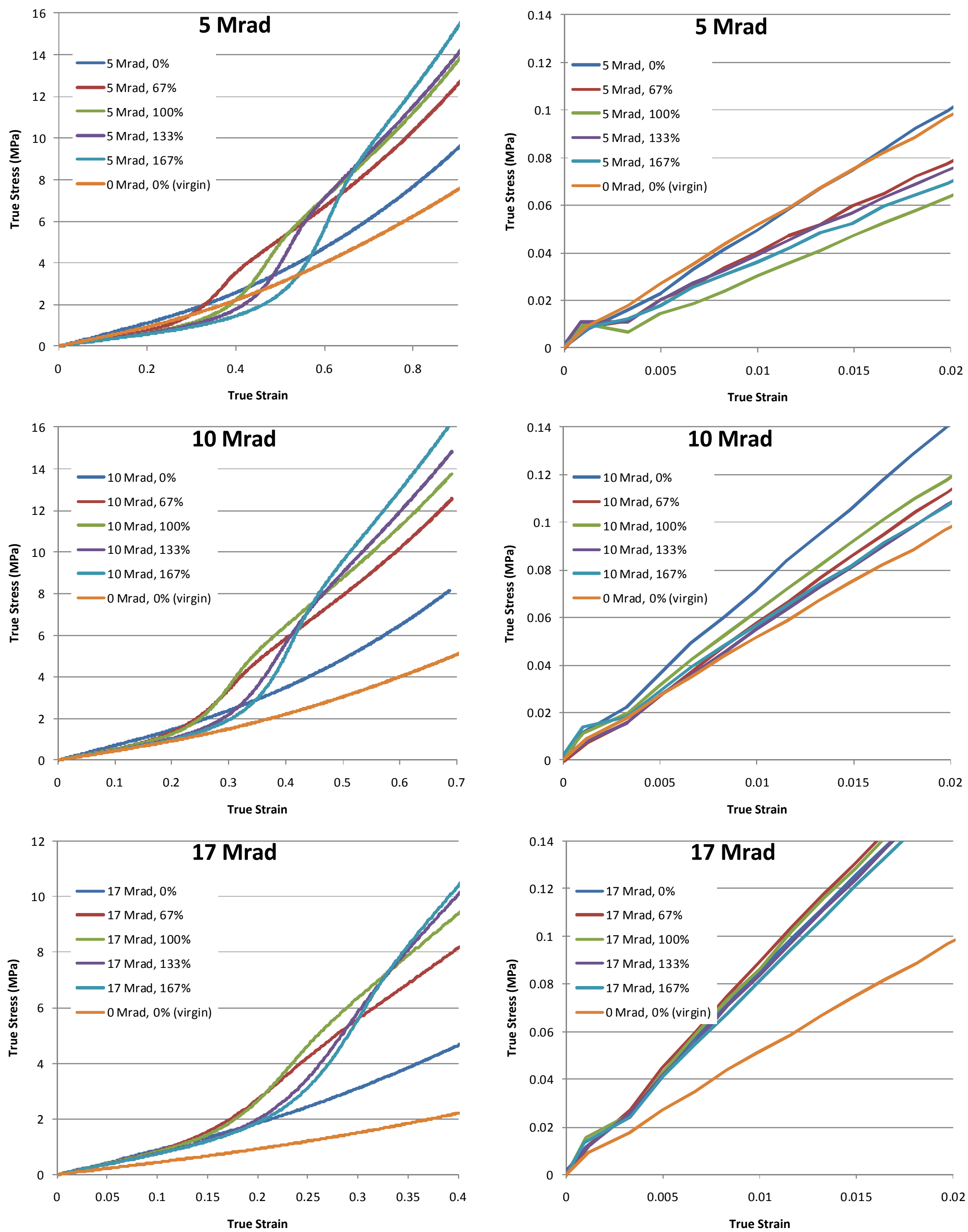

Fig. 3. True stress-strain curves from the loading segment of the first cycle for TR-55 specimens irradiated under tensile strain (plots on left). Plots of the low strain region used to calculate the modulus are shown on the right. A curve representing the virgin material is also shown. Specimen 2 data is shown.

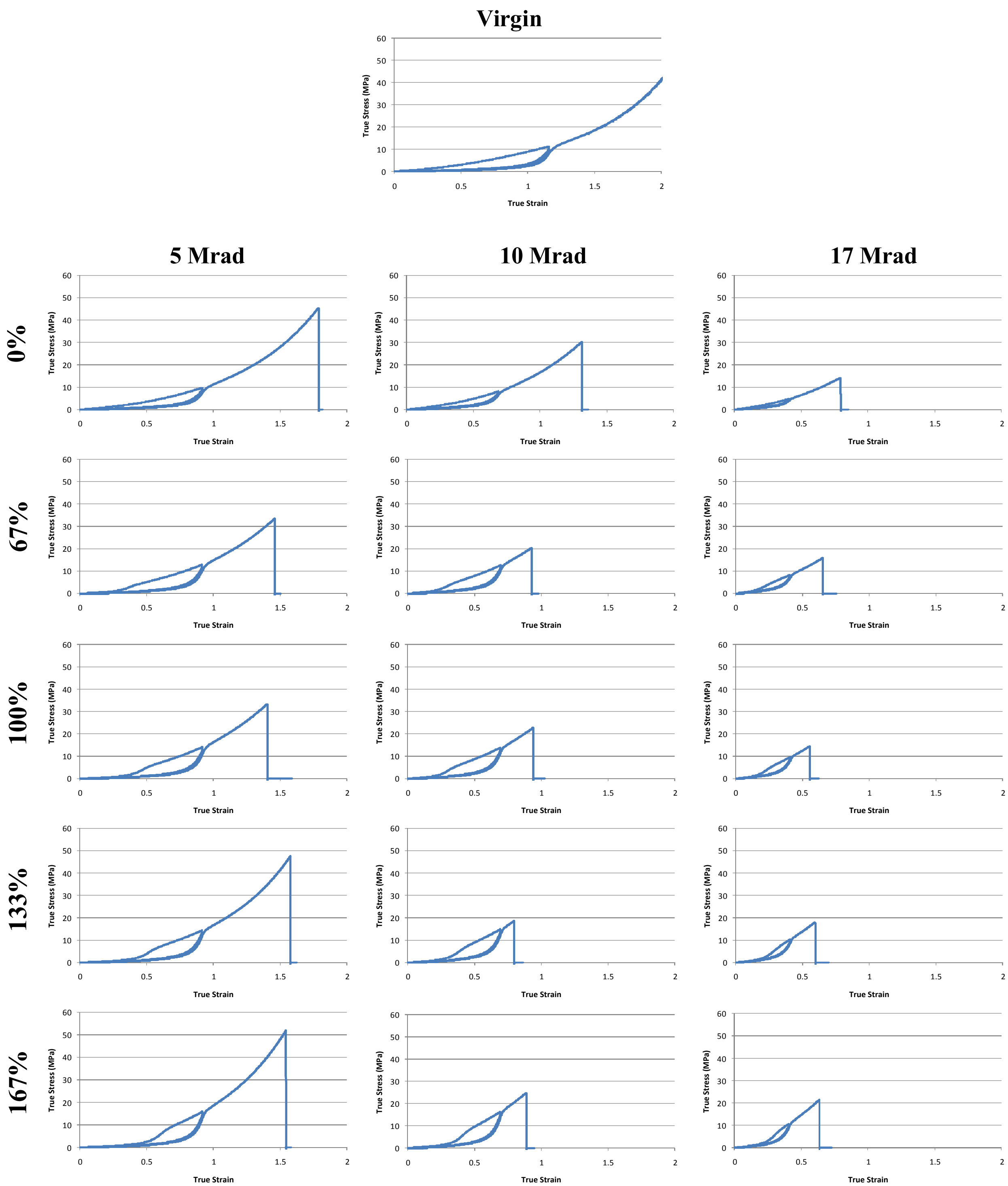

Fig. 4. True stress-strain curves of the four load/unload cycles and subsequent strain to failure for virgin and aged TR-55 specimens. The maximum engineering strain of the first four cycles was 2.2 for the virgin specimen and 1.5, 1.0, and 0.5 for the 5, 10, and 17 Mrad irradiated specimens, respectively. Specimen 2 data is shown.

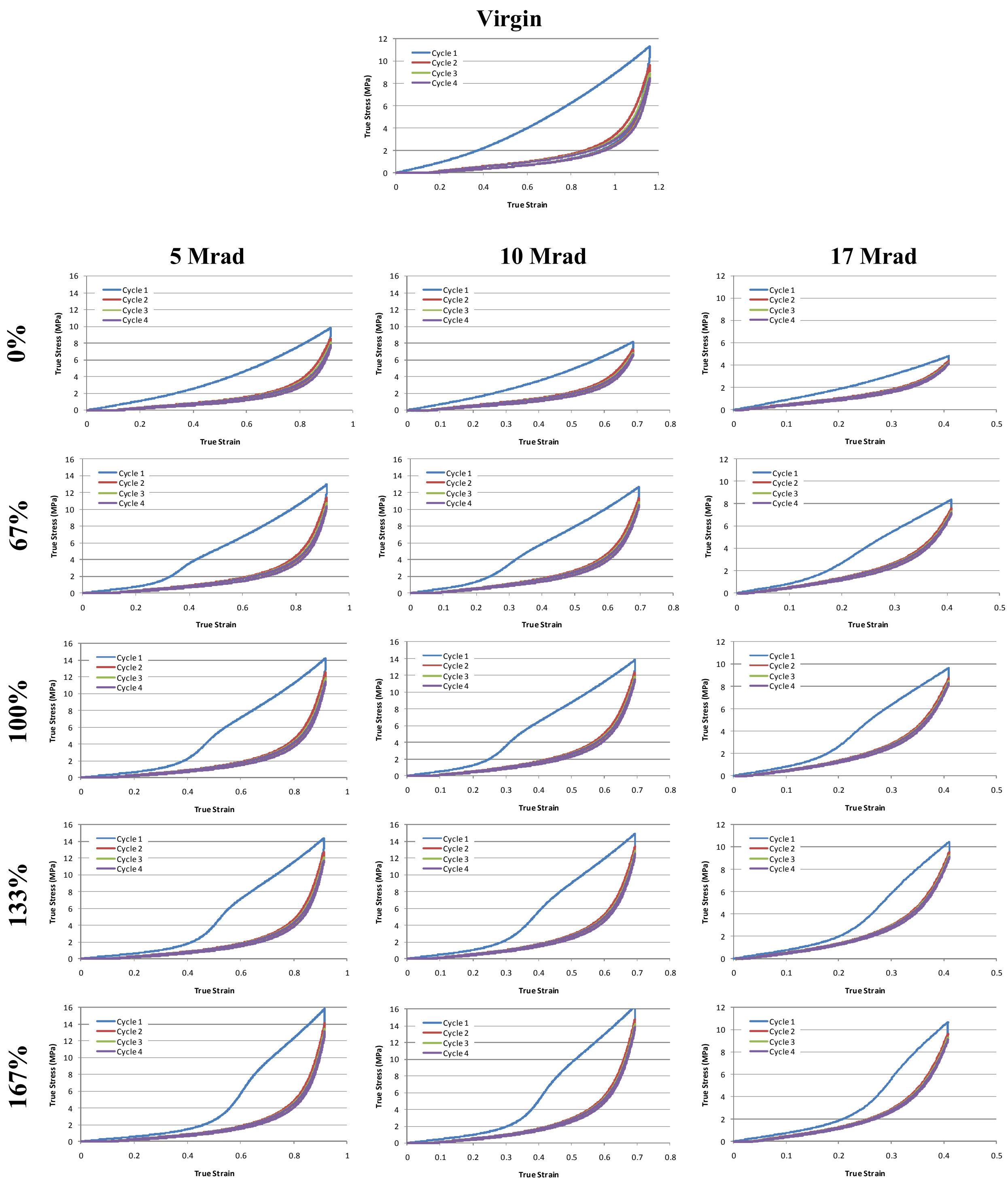

Fig. 5. True stress-strain curves of the four load/unload cycles for virgin and aged TR-55 specimens. The maximum engineering strain was 2.2 for the virgin specimen and 1.5, 1.0, and 0.5 for the 5, 10, and 17 Mrad irradiated specimens, respectively. Specimen 2 data is shown.

APPENDIX A
Strain Aging Process

The stretching fixture consisted of two movable clamps on a base plate with preset pins to determine the separation between the clamps. With the clamps spaced 1.5" apart (see Fig. A1), a rectangular strip (~10 mm wide and <1 mm thick) of TR-55 was secured in the fixture (i.e., original specimen length L_0 = 1.5"). To stretch the specimen, the separation between the clamps was increased to one of four lengths, L_{aging}:

#1: 2.5" (67% elongation, λ_1 = 1.67)
#2: 3.0" (100% elongation, λ_1 = 2.00)
#3: 3.5" (133% elongation, λ_1 = 2.33)
#4: 4.0" (167% elongation, λ_1 = 2.67)

The axial stretch ratio λ_1 is given by

$$\lambda_1 = L_{aging}/L_0$$

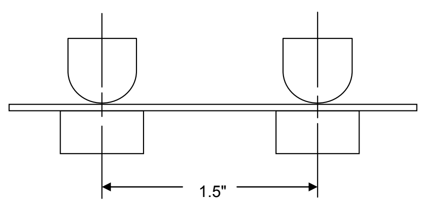

Fig. A1. Schematic of a specimen clamped in its initial pre-stretched position.

To determine the extent of specimen slippage in the clamps (before irradiation), the actual stretched length L_{aging} was calculated for each specimen (Table A1) based on the measured change in specimen width for an incompressible elastomer according to Chinn et al., Polymer Degradation and Stability 2006; 91:555-64:

$$L_{aging} = \frac{L_0}{\left(w_{aging}/w_0\right)^2}$$

where L_0 is the unstrained specimen length (1.5") and w_0 and w_{aging} are the widths of the specimen in the initial (unstrained) and strained states, respectively.

Table A1: Calculated Specimen Length in Stretched Position

Nominal	**5 Mrad**			**10 Mrad**			**17 Mrad**		
L_{aging}	**Aging Date**	**L_{aging}**	**λ_1**	**Aging Date**	**L_{aging}**	**λ_1**	**Aging Date**	**L_{aging}**	**λ_1**
#1: 2.5" (λ_1=1.67)	1/7/2010	2.46"	1.64	1/21/2010	2.48"	1.66	1/13/2010	2.44"	1.63
#2: 3.0" (λ_1=2.00)	1/7/2010	2.89"	1.93	1/21/2010	2.92"	1.95	1/13/2010	2.96"	1.97
#3: 3.5" (λ_1=2.33)	1/7/2010	3.24"	2.16	1/21/2010	3.30"	2.20	1/13/2010	3.36"	2.24
#4: 4.0" (λ_1=2.67)	1/7/2010	3.80"	2.54	1/21/2010	3.76"	2.51	1/13/2010	3.77"	2.51

APPENDIX B

Stress and Strain Calculation

The following formulas were used to calculate stress σ and strain ε. Formulas for engineering and true (Cauchy) values are shown.

L_0 = initial specimen length (i.e., initial grip separation)
L = specimen length during tensile testing
A_0 = initial cross-sectional area of specimen
A = cross-sectional area of specimen during tensile testing
w_0 = initial specimen width
t_0 = initial specimen thickness
F = measured load during tensile testing

$$\varepsilon_{\text{eng}} = \frac{L - L_0}{L_0}$$

$$\varepsilon_{\text{true}} = \ln\left(1 + \varepsilon_{\text{eng}}\right) = \ln(L/L_0)$$

$$\sigma_{\text{eng}} = \frac{F}{A_0} = \frac{F}{w_0 t_0}$$

$$\sigma_{\text{true}} = \frac{F}{A} = \frac{F}{A_0 L_0 / L} = \frac{FL}{w_0 t_0 L_0}$$

The following formulas were used to calculate the theoretical Hookean and neo-Hookean true stress at low strain based on the Young's modulus E given by the initial slope of the true stress-strain curve.

$$\sigma_{\text{Hookean}} = E\varepsilon_{\text{true}}$$

$$\sigma_{\text{neo-Hookean}} = \frac{E}{3}\left(\lambda - \frac{1}{\lambda^2}\right), \text{ where } \lambda = L/L_0$$